# Precipitated Silica from Sodium Silicate by CO 2 on Fixed Bed Column

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# Precipitated Silica from Sodium Silicate by $CO_2$ on Fixed Bed Column

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**Keywords:** bagasse ash, carbon dioxide, fixed bed, sodium silicate, precipitated silica

**Abstract.** The precipitated silica prepared by reaction of sodium silicate and gas $CO_2$ on fixed bed column have been production successfully. In this study, silica from bagasse was extraction by sodium hydroxide 2N solution to produce sodium silicate solution. The sodium silicate solution was dilute by demineralize water to produce some concentration in the range of 0.33-0.98 %$SiO_2$. Fixed bed column has a diameter of 7.5 cm with a height of 50 cm and a pH control apparatus. $CO_2$ gas and sodium silicate liquid are both flowed from under of the column with a specified flow rate. The precipitate process was carried out on a fixed bed column with high of bed in the range of 10-30 cm. The effect of silica concentration and the high of the bed on characterize of the precipitated silica product have been studied. The precipitated silica product characterized by XRF, XRD, SEM-EDX and BET. The quality of precipitated silica produced in the range concentration of 95-98 w% $SiO_2$, surface area (BET) in the range of 46.1 – 58.8 $m^2/g$.

## Introduction

The precipitated silica is required to support various types of industrial operations such as vehicle, rubber, cosmetics, electronics, agriculture and other industries. The grade quality of precipitate silica products depends on the type of industrial users. The production of precipitated silica starts with the reaction of an alkaline silicate solution, usually but not necessarily sodium silicate solution, with various types of acids such as hydrochloric, sulfuric, acetate and carbon dioxide [1,2]. Preparation of sodium silicate by alkali extraction has been successfully used to recover silica from bagasse ash, geothermal sludge and rice hull ash to produce porous silica powder [3,4,5,6]. Sodium silicate was used in foundries to bind sand grains using carbon dioxide ($CO_2$) directly where the hardening of the sand immediately occurs as a result of the chemical reaction between sodium silicate and carbon dioxide[7]. Cai et al [8] was reported a method for preparation of silica powders using sodium silicate and carbon dioxide by pressured carbonation as a precipitating agent. The reaction time, temperature and concentration of sodium silicate were influential on silica powder characterize. Combination of a microreactor and a stirred reactor for production of a large-pore-volume and narrow-pore-diameter distribution of silica materials was reported [9] while the gelatine time was controlled by improing the mixing performance.

In the previous study reaction of sodium silicate from bagasse ash with $CO_2$ was provide in the bubble column. The duration of precipitation, the addition rate of reactants, sodium silicate concentration, and pH can vary the properties of the silica [10]. The residence time and the rate of $CO_2$ gas was an obstacle in the formation of precipitated silica. This study developed the formation of precipitated silica from reaction of sodium silicate and $CO_2$ on fixed bed column in order to increase the contact time in the column. The existence of bed material in the column was necessary to be studied considering the formation of precipitated silica will be able to inhibit the subsequent precipitation process and inhibit the performance of the fixed bed column itself too. By studying the residence time with the rate of gas controller in a column it is expected that the precipitated material can flow with liquid before deposited in the bed column. The precipitation silica product prepared by acidification proses was followed a two-step process, namely the formation of primary particles followed by flocculation which expand exponentially with an increase in acid concentration and

with salt accelerates the process [7,11]. The reaction of sodium silicate solution with carbon dioxide besides producing precipitated silica would also cause sodium carbonate salt. The presence of salt trapped in precipitated silica particle can affect the purity and the surface area of the silica product. In addition to investigate the fixed bed column performance for the formation of precipitated silica at various height bed, this study also analyzed the morphology of the precipitated silica product characteristic.

## Materials and Methods

Silica source from bagasse ash obtained from the sugar industry. Sodium hydroxide (NaOH) as solvent on silica extraction obtained from CV. Bratachem Surabaya and carbon dioxide ($CO_2$) gas as precipitator obtained from CV Medica Vanjaya Surabaya. The bagasse that obtained from the sugar industry waste is black in color indicate that bagasse still contains a lot of carbon

The production precipitated silica from sodium silicate in this research was follow the two steps process namely 1) preparation of sodium silicate solution from bagasse ash 2) preparation of precipitated silica from sodium silicate and $CO_2$ gas in fixed bed column.

The Bagasse ash from sugar industry burned on furnace at 700 C for 2 h. After burning in the furnace bagasse ash was then cooled and grinded to a size about 80-100 mesh and analyzed for its silica content. The chemical composition of bagasse ash by XRF analyze was $SiO_2$: 73%, CaO: 8.63%, $K_2O$ 5.01%. The extraction of silica prepared by extraction of 80g bagasse ash using 1000ml of sodium hydroxide (NaOH) 2 N solution at 95 °C for 2 h to produce the sodium silicate solution. The sodium silicate solution was analyzed for the content of sodium oxide ($Na_2O$) with AAS method and silica content ($SiO_2$) by Spectrophotometry method. The result showed that sodium silicate solution was has a silica concentration of 4.85 % and sodium oxide concentration of 4.57 %. Sodium silicate solution was then dilution by demineralized water in the range ratio of 1:1; 1:2; 1:3; 1:4; and 1:5 obtained the concentrations of 0.33; 0.39; 0.49; 0.65 and 0.98% $SiO_2$ by stoichiometry calculation method.

The Sodium silicate solution is pumped by dosage pump with a flow rate of 60 ml/min into the fixed bed column (diameter column of 7.5 cm and height of 50 cm) and followed by injection of carbon dioxide ($CO_2$) gas with a rate of 4 l /min. The sodium silicate solution will react with carbon dioxide gas, pH of sodium silicate solution decrease from value of 12 to 7. The precipitated silica formed in the column will also flow with the liquid to the top of the column and then flow into the settling tank until reached a volume of 2 liters and value of pH adjusted 7. From the settling tank, precipitated silica was filtered using whatman 41 paper filter then washed and dried in an oven at 100 °C for 18h. Precipitated silica from oven was pounded to a size about 100 mesh for analysis purpose.

In this study the reaction of sodium silicate solution with carbon dioxide ($CO_2$) gas was followed the reaction:

$$Na_2SiO_3 + CO_2 + H_2O \rightarrow SiO_2 + Na_2CO_3 + H_2O \quad (1)$$

The precipitated silica product was characterize by the X-ray fluorescence (XRF), X-ray diffraction (XRD), surface area Brunauer-Emmet-Teller (BET) and Scanning electron microscopy with energy diffraction x-ray (SEM-EDX ).

## Result and Discussion

Fig. 1 showed the diffraction pattern of precipitated silica product prepared by sodium silicate concentration of a) 0.49, 0.65 and 0.98 %$SiO_2$ before washing, and b) 0.33-0.98 % $SiO_2$ after washing. Based on the XRD results for three samples (0.49, 0.68, and 0.98%) which have high silica concentrations, they indicated the presence of salt ($Na_2CO_3$) impurities as shown in Fig. 1a, then all samples (0.33-0.98 % $SiO_2$) were washed in order to remove the impurities (Fig. 1b). It can be stated that after the washing process in these samples the product's purity level also increases.

The subsequent analysis of BET, SEM-EDX and XRF was based on precipitated silica after washing.

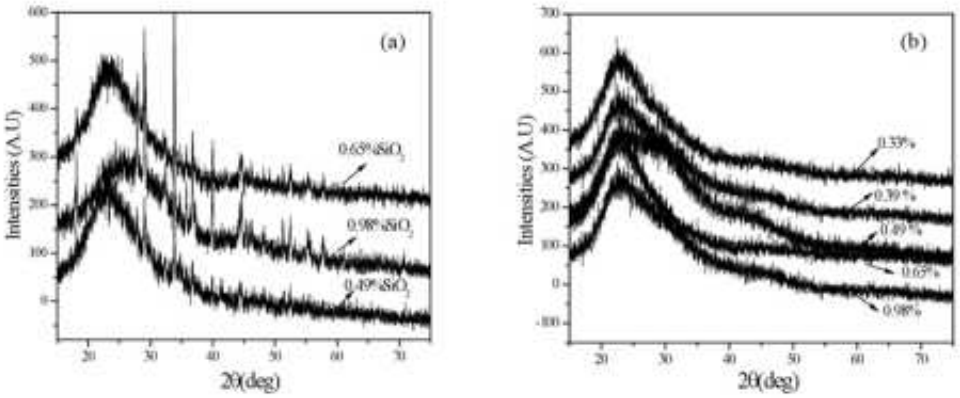


Fig. 1. Diffraction pattern of precipitated silica prepared (a) before washing and (b) after washing.

Fig. 2 showed the effect of the height of bed on a) concentration of precipitated silica and b) amount of precipitated silica. Silica concentration increases with bed height. The higher the bed height is from 10 to 30 cm in a fixed bed column, the contact time between gas and liquid will be longer, as a result the greater the amount of precipitated silica formed. However, if the bed is too high, the residence time that is too long will cause sedimentation from precipitated silica so that it can inhibit the reaction rate and inhibit the performance of fixed bed columns. The amount of silica precipitation product produced at bed height from 10 to 30 cm and silica concentration from 0.33 to 0.98 % is shown in Fig. 2b. The amount of silica precipitation obtained depends on the silica concentration in sodium silicate used. The greater the concentration of sodium silicate from 0.33 to 0.98 %, the greater the amount of silica obtained from about 10 to 33 g. However, the use of low sodium silicate concentration (0.33%) can produced the least amount of precipitated silica (about 10-17g) but its purity is highest (up to 99%). However, in obtaining a large amount of silica precipitation it is obtained a lower concentration of silica.

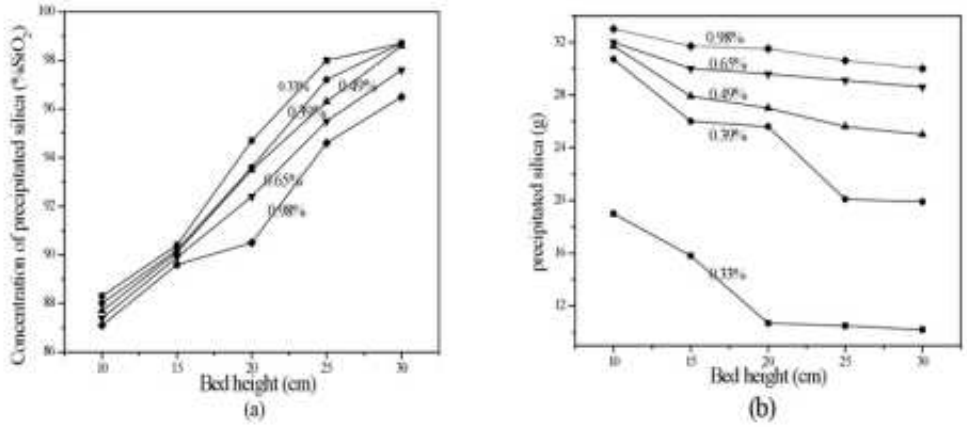


Fig. 2. The effect of height of bed on (a) concentration of precipitated silica and (b) amount of precipitated silica.

Fig. 3 showed the typically isotherm of adsorption-desorption of precipitated product prepared by sodium silicate concentration of 0.98% and 0.33% SiO2. The isotherms can be identified as type V isotherms according to the IUPAC classification, which are correspond to micro porous solids. The hysteresis can be classified as a type H2, in which the hysteresis loops of the desorption branch are steeper than those of the adsorption branch. T the hysteresis loops was characterize of bottlenec pores and small particles. The precipitated silica prepared by sodium silicate 0.33% $SiO_2$ have a surface area (BET) of 58.811 $m^2/g$, pore volume of 0.263 cc/g d pore diameter of 3.781 nm, while the precipitated silica prepared by sodium silicate 0.98% have a surface area (BET) of 46.089 $m^2/g$, pore volume of 0.160 cc/g and pore diameter of 4.261 nm.

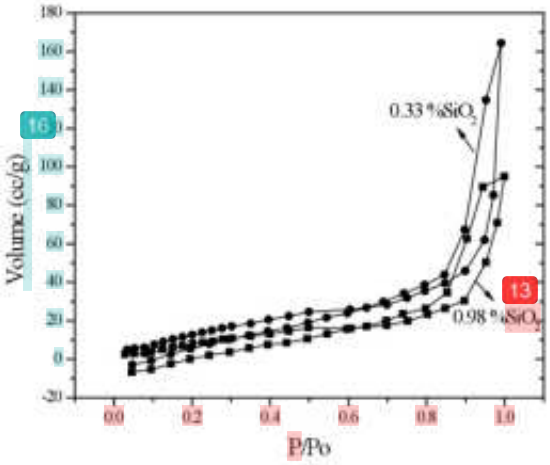


Fig. 3. Adsorption-desorption isotherm of precipitated silica.

Fig. 4 showed the SEM ima of precipitated silica prepared by sodium silicate 0.49% $SiO_2$ by height of bed in column was a) 10 cm b) 20 cm and c) 30 cm. Particles of precipitated silica preparation on a bed height of 10 cm have a random shape of particle, while precipitated silica preparation on a bed height of 30 cm obtained the particles with the shape almost spherical and uniform in size.

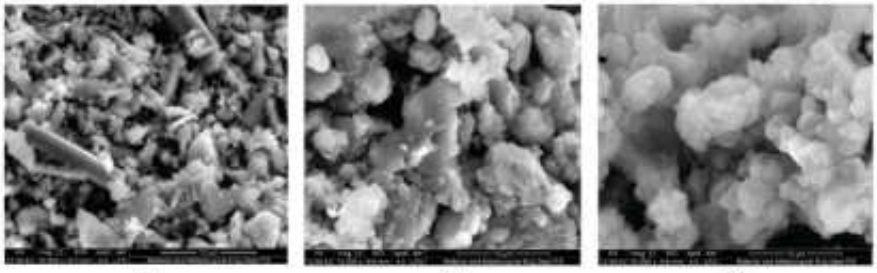


Fig. 4. SEM images of precipitated silic repared by sodium silicate 0.49% $SiO_2$ with height of bed on column of a) 10 cm b)20 cm and c) 30 cm.

The microanalysis by EDX showed that precipitated silica prepared by sodium silicate 0.49 % the magnification particle size in the range of 1-20μ the component of silica was increased from 28.58 to 42.53 %. Meanwhile, precipitated silica prepared by sodium silicate 0.39 % and 0.33 % in the range of particle size of 1-20 μm the component of silica decrease from 44.29 to 37.32%, and 47.67 to 43.88% respectively.

**Summary**

Precipitated silica particles were successfully produced from sodium silic using precipitator $CO_2$ on fixed bed column. The concentration of silica in product tended to increase with the increasing of bed height. The morphology of precipitated silica prepared on bed height of 30cm was more uniform and almost spherical in shape. The surface area of precipitated silica was reached 58.811$m^2$/g on fixed bed column with a diameter of 7.5 cm, height of 15 cm and bed height of 30 cm.

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